This invention relates to the separation of oil from bituminous sands such as Athabasca tar sands. More particularly the invention pertains to a process involving a hot water separation step for primary recovery of oil from the bituminous sands and a separate recovery step for recovering oil which fails to separate from the water layer or middlings derived from the primary separation step.

Deposits of oil-containing sands generally referred to as bituminous sands or tar sands occur in various areas throughout the world. The largest known deposits are the tar sands found in the Athabasca region of northern Alberta Province, Canada, which extend over an area of many square miles and range in thickness up to more than 200 feet. Such bituminous sands generally have oil contents in the range of 5-20% by weight. The mineral matter not only includes the sand component but also usually comprises silt and clay components consisting of fine particles having diameters of less than 44 microns. In any particular deposit the amount of the silt and clay components can vary from place to place, generally ranging from 1 to 50%, more usually 10-30%, by weight based on the mineral content only. The bituminous sands also usually contain a small amount of water, e.g., 1-10% by weight, in the form of a film around the sand grains.

Numerous methods have been proposed heretofore for separating oil from bituminous sands such as Athabasca tar sands. The best known methods often are referred to in the art as the "hot water method" and the "cold water method". These procedures have been described in Proceedings, Athabasca Oil

Sands Conference, September, 1951. In the hot water method the bituminous sands are jetted with steam and mulled with a minor amount of hot water at a temperature typically of 176°F., and the pulp is then dropped into a turbulent stream of circulating hot water and carried to a separation cell maintained at a temperature of about 185°F. In the separation cell sand settles to the bottom, oil rises to the top in the form of a froth and an aqueous middlings layer comprising clay and silt is formed between these layers. In limited instances, this procedure will give a reasonably good separation of oil from tar sands which have low clay and silt contents; however for tar sands having high clay and silt contents a good separation usually is not obtained and large amounts of the oil remain with the middlings layer. Even for sands of low clay and silt content an appreciable amount of the oil often fails to separate out as froth and remains dispersed in the middlings layer.

The cold water method, in which the separation is conducted at a temperature of 73-81°F., requires the use of a light hydrocarbon solvent, such as naphtha or kerosene, in order to achieve a reasonably good separation. This use of a solvent is objectionable, since it requires the provision of vapor tight equipment and also considerable amounts of solvent tend to be lost with the sand tailings. For a commercial operation it is highly desirable to avoid the use of any solvent in the separation step.

The present invention provides an efficient process for separating oil from bituminous sands which is capable of effecting good recovery of the oil or tar from the sands regardless of the clay and silt contents of the feed material. The process also does not require the use of any solvent in the separation zones. The invention can be considered as an improvement in the hot water process as heretofore proposed.

The invention comprises the combination of a hot water separation step for primary recovery of oil from the sands together with a scavenger step for further treatment of the middlings layer obtained from the primary separation step to recover an additional amount of oil therefrom; and it further comprises regulating the amount of water introduced to the primary separation step and the rate of transfer of middlings layer therefrom to the scavenger step so as to maintain the density and/or viscosity of the middlings layer as hereinafter specified. In one embodiment the process involves the following procedure:

(1) forming a pulp of the bituminous sands with a minor amount of water in a pulping zone while heating the mixture with steam;

(2) removing pulp therefrom and mixing the same with hot water and the hereinafter specified recycle stream in a dilution zone, the amount of said hot water plus the water added to the bituminous sands in forming said pulp being 0.2-3.0 lbs./lb. of the bituminous sands;

(3) flushing the mixture from the dilution zone into a separation zone;

(4) settling the mixture in the separation zone at a temperature in the range of 130-210°F., more preferably 170-210°F., to form an upper oil froth layer, a middlings layer comprising water, clay and oil, and a sand tailings layer;

(5) separately removing the oil froth layer and the sand tailings layer;

(6) removing a stream of middlings layer from the separation zone and passing it to the dilution zone as the aforesaid recycle stream;

(7) passing a second stream of middlings layer to a scavenger zone and therein subjecting it to air flotation to recover an additional amount of oil froth;

(8) regulating the amount of water incorporated with the bituminous sands in Step (1) and the rate of passage of the second stream to the scavenger zone in Step (7) to regulate and maintain the density of the middlings layer within the range of 1.03-1.50 g/cc and/or the viscosity thereof within the range of 0.5-10 centipoises; and

(9) removing from the scavenger zone and discarding from the system middlings material of depleted oil content comprising clay dispersed in water.

The invention is more specifically described in conjunction with the accompanying drawing which presents a schematic illustration of the present process.

Referring to the drawing the bituminous oil sand is fed to the system through line 10 where it first passes to a conditioning drum or muller 11. Water is fed to the muller via line 12 and steam is introduced thereto through line 13. The

total water so introduced in liquid and vapor forms is a minor amount based on the weight of the tar sands processed and generally is in the range of 10-45% by weight of the mulled mixture. The conditioning drum 11 is provided with suitable kneading or mixing means (not shown) to give the desired mulling action. Enough steam is introduced through line 13 to raise the temperature in the conditioning drum to within the range of 130-210°F. and preferably to above 170°F. Mulling of the tar sands produces a pulp which then passes from the conditioning drum as indicated by line 14 to a screen indicated at 15. The purpose of screen 15 is to remove from the tar sands pulp any debris, rocks or oversized lumps as indicated generally at 16.

The conditioned tar sands pass from screen 15 to a pulp box 17 which serves as a zone for diluting the pulp with additional water before passage to primary separation zone 18. Hot water from heater 27 is passed through line 19 to pulp box 17 and additional steam is fed thereto through line 20 if necessary to maintain the temperature in the range of 130-210°F. and preferably above 170°F. Also a middlings stream which is withdrawn from the primary separator 18 is recycled through lines 21 and 19 to the pulp box. This recycle stream serves to provide sufficient liquid to flush the tar sands pulp from the pulp box and effect transfer of the pulp to the separator. Another important function of the recycle stream is to cause dispersion of the pulped material as it is fed into separation zone 18. However, such recycling of middlings is not essential in all cases, particularly when the clay content of the tar sands is high. In this event a relatively high rate of fresh feed water introduction through heater 27 can be employed to compensate for the high clay content while a correspondingly high rate of transfer of middlings layer through line 26 as hereinafter described can be maintained; and under these circumstances

recycling of the other stream of middlings through lines 21 and 19 to pulp box 17 is not required.

Modifications that may be made in the process as above described include sending a minor portion of the middlings recycle stream from line 21 through a suitable line (not shown) to muller 11 to supply all or a part of the water needed therein other than that supplied through condensation of the stream which is consumed. Also, if desired, a stream of the middlings recycle can be introduced onto the screen 15 to flush the pulp therethrough and into pulp box 17.

Separation zone 18 can comprise a large cylindrical or rectangular tank, or battery of tanks, which may, if desired, be provided with heating coils 22 for maintaining a temperature in the range of 130-210°F., and preferably above 170°F. The separator is supplied with an oil froth withdrawal line 23 adjacent the top and a sand tailings removal line having star valve 24 or any other suitable control discharge means at the bottom. Separator 18 also has an intermediate withdrawal line 26 through which a stream of middlings layer is removed in addition to that recycled through line 21.

In operation of the process the pulped tar sands are continuously flushed from pulp box 17 through line 25 into separator 18 by means of the relatively large flow of water supplied by the middlings recycle stream and the fresh water from heater 27. The settling zone in separator 18 is relatively quiescent so that oil froth rises to the top and sand settles to the bottom. The froth results by virtue of air or gas being trapped in the oil sands during the previous conditioning operations and thus imparting to the oil phase an effective density considerably below that of water. The separation zone 18 should

have a horizontal cross-sectional area such that there are from 0.1 to 10 sq. ft. of cross-sectional area per ton of virgin tar sands per hour that passes through screen 15 (calculated on a dry basis). More preferably such area should be in the range of 0.5-3.0. Further, the volume of the separation zone should be such that the average retention time of the pulped tar sands charged will be 1-60 minutes, more preferably 2-20 minutes. The froth is continuously or intermittently removed from the top of the separation zone through line 23, and sand tailings are removed from the bottom and discarded.

The middlings layer obtained in separation zone 18 will contain most of the silt and clay which was present in the tar sands in their natural state. In order to prevent the build up of clay in the system it is necessary to continually discard some of the middlings layer and supply enough water in the conditioning operations to compensate for that so discarded. The rate at which the middlings needs to be removed from the system depends upon the content of clay and silt present in the tar sands feed and this will vary from time to time as the content of these fines varies. If the clay and silt content is allowed to build up in the system, both the density and the viscosity of the middlings layer will increase. Concurrently with such increase an increase in the proportions of both the oil and the sand retained by the middlings will occur. If the clay and silt content is allowed to build up too high in the system, effective separation no longer will occur and the process will become inoperative. Hence it is important to regulate the withdrawal of middlings through line 26, and the addition of fresh water to the system to compensate for water thus removed, in a way that will keep the separation step operating properly. However, even when this separation step is operating in an optimum

manner, the middlings layer withdrawn through line 26 will contain a substantial amount of oil which did not separate. Hence the middlings layer withdrawn through line 26 is, for purpose of description, herein referred to as "oil-rich middlings".

In practicing the invention the rate of addition of fresh water to the system and the rate of removal of middlings layer from separation zone 18 through line 26 are regulated in accordance with either the density or the viscosity of the middlings layer or both. When density is used for the control, such addition and removal are carried out so that the middlings density is maintained in the range of 1.03-1.50 g/cc, more preferably 1.10 to 1.20 g/cc. It is preferred, however, to utilize viscosity to effect the control, in which case the water addition and removal are carried out to maintain the middlings viscosity in the range of 0.5 to 10 centipoises, more preferably 0.6 to 3.0 centipoises. Periodic or continuous measurements of either viscosity or density for the middlings phase can be made, and the removal of middlings through line 26 and corresponding addition of fresh water to the system can be regulated in accordance with the measured values to maintain the value within the range desired. Whenever either density or viscosity tends to become higher than is desired, an increase is made in the rate of middlings removal and corresponding rate of fresh water addition; and if density or viscosity values tend to become too low, decreases in these removal rates are effected.

When density is the variable used for control of the process as above described, the density of the middlings layer should be measured at the operating temperature and without permitting any solids to separate from the middlings prior to testing. A convenient procedure is to collect middling samples

periodically and immediately test each sample by means of a pycnometer. Alternatively a continuous sampling stream of middlings can be passed through a density monitoring device to obtain continuous automatic density determinations. When viscosity is employed as the control variable, the middlings to be tested should be maintained at the operating temperature and allowed to settle for 5 minutes, the tar and sand that has settled out should be removed, and the resulting middlings layer should be used for the viscosity determination. This can be measured at the operating temperature by means of a Brookfield Syncho-Lectric viscometer with an adapter having a cylindrical spindle (0.99" diameter and 3.5" length) mounted in an open-end tube (1.09" I.D.) and rotated at 60 r.p.m. This type of viscometer has been described by Van Wazer et al. in the textbook "Viscosity and Flow Measurement", pages 139-150 (Interscience Publishers, 1963). For the purpose of the invention the density and viscosity values within the above-specified ranges which are utilized for controlling the process are determined in the aforesaid ways or in any other ways which give essentially equivalent values.

As a general rule the total amount of water added to the natural bituminous sands as liquid water and as steam prior to the separation step should be in the range of 0.2-3.0 lbs./lb. of the bituminous sands. The amount of water needed within this range increases as the silt and clay content of the bituminous sands increases. For example, when 15% by weight of the mineral matter of the tar sands has a particle size below 44 microns, the fresh water added generally can be about 0.3-0.5 lb./lb. of tar sands. On the other hand when 30% of the mineral matter is below 44 microns diameter, generally 0.7-1.0 lb. of water should be used per pound of tar sands. Correspondingly the amount of

oil-rich middlings removed through line 26 will vary depending upon the rate of fresh water addition. As a general rule the rate of withdrawal of oil-rich middlings through scavenger zone 29 will be 10-75 gallons per ton of tar sands processed when 15% by weight of the mineral matter is below 44 microns and 150-250 gallons per ton when from 25-30% of the mineral is of this fine particle size.

As previously mentioned, the middlings layer withdrawn through line 26 will still contain a substantial amount of oil even though the separation step is operated under optimum conditions. The amount of oil remaining in the middlings layer appears to be more or less related to the percentage of clay and/or silt present in the tar sands being processed, varying directly with the amount of clay and/or silt present. For example, typical oil recovery values for the froth from tar sands in which 15% of the mineral matter is less than 44 microns and from sands in which 25-30% is less than this size are respectively, 85% and 60%. For commercial operation it is highly desirable to obtain increased recoveries over such values as these which are obtainable heretofore by the hot water process. This is particularly true when the tar sands as mined contain a relatively high proportion of clay and silt components.

In accordance with the invention the oil-rich middlings stream withdrawn from separator 18 through line 26 is sent to a scavenger zone 29 wherein an air flotation operation is conducted. It has now been found that air flotation is an effective way for recovering oil which has failed to separate from the middlings layer. By way of example, the flotation step will increase the total oil recovery typically to 90-95% for tar sands in which 15% of the mineral matter is less than 44 microns and to 80-90% for those in which 25-30% of the mineral matter is less than 44

microns. In a large size commercial operation an increase of oil recovery of even a few percentage values can amount to a large volume of additional oil per day. Hence the present invention provides a distinct improvement over the hot water process as heretofore proposed for commercial practice.

The processing conducted in scavenger zone 29 involves air flotation by any of the air flotation procedures conventionally utilized in processing of ores. This involves providing a controlled zone of aeration in the flotation cell at a locus where agitation of the middlings is being effected so that air becomes dispersed in the middlings in the form of small bubbles. The drawing illustrates a flotation cell of the sub-aeration type wherein a motorized rotary agitator 30 is provided and air is fed thereto in controlled amount as by means of line 31. Alternatively the air can be sucked in through the shaft of the rotor. The rotor effects dispersion of the air in the middlings. This air causes the formation of additional oil froth which passes from the scavenger zone 29 through line 32 and thence to line 23 for further processing in admixture with the froth derived from the primary separation in zone 18. The residence time in scavenger cell 29 can vary widely but generally is in the range of 1-60 minutes and usually 2-20 minutes. An oil-lean middlings stream is removed from the bottom of scavenger zone 29 via line 33 and is discarded from the process. The oil-lean middlings contains a substantial proportion of the clay and silt components that were present in the original tar sands, and discarding thereof from the process prevents the build up of this fine material in the separation zone 18. The amount so discarded is such as to maintain the viscosity and density of the oil-rich middlings in zone 18 within the ranges as specified hereinbefore.

The mixed froths from lines 23 and 32 will contain some water and an appreciable amount of the finer mineral matter that was present in the tar sands. Generally this material will be sent to a processing zone (not shown) wherein the water and mineral matter are removed. This can be done by diluting the froth with naphtha and treating the mixture in an electrostatic precipitator or in centrifuges to effect dehydration and demineralization.

For securing optimum results with the process as above described an alkali metal-containing alkaline reagent generally should be added to the conditioning drum usually in amount of from 0.1 to 3.0 lbs. per ton of tar sand. The amount of such alkaline reagent preferably is regulated to maintain the pH of the middlings layer in separator zone 18 within the range of 7.5-9.0. Best results seem to be obtained at a pH value of 8.0-8.5. The amount of the alkaline reagent that needs to be added to maintain a pH value in the range of 7.5-9.0 may vary from time to time as the composition of the tar sands as obtained from the mine site varies. The best alkaline reagents to use for this purpose are caustic soda, sodium carbonate or sodium silicate, although any of the other alkali metal-containing alkaline reagents can be used if desired.

The following example illustrates the invention more specifically:

The invention is utilized to recover the oil from Athabasca tar sands containing by weight about 10% bituminous matter and 89% mineral matter. Twenty-five percent of the latter consists of particles having diameters less than 44 microns. On an hourly basis 100 lbs. of the tar sands, 0.1 lb. of concentrated caustic soda solution, 30 lbs. of water and

steam are fed into a conditioning drum and the mixture is heated to about 180°F. while being mulled. The resulting pulp is passed through a screen and then to pulp box 17 as shown in the drawing. Hot water at 190°F. in amount of 74 lbs. and a middlings recycle stream in amount of 200 lbs. are passed through line 19 into the pulp box and the mixture continuously flushes into separator 18 wherein the temperature is maintained at 190°F. The pH of the middlings is held at about 8.4 by the addition of caustic soda at the rate above specified. From the separator sand tailings are removed at a rate of 78 lbs./hr. from the bottom and froth is removed from the top at a rate of about 10 lbs./hr. The tailings are composed of about 74% mineral matter, 25% water and 1% oil by weight. The froth is approximately composed by weight of 50% oil, 10% mineral matter and 40% water. The oil content thereof corresponds to a recovery of about 50% of the oil in the original tar sands. A stream of oil-rich middlings in amount of 112 lbs./hr. and composed of about 4 lbs. of oil, 27 lbs. of mineral matter and 81 lbs. of water is withdrawn from separator 18 and is transferred to a scavenger zone wherein it is subjected to air flotation in a subaeration type air flotation cell 29. This rate of transfer of the oil-rich middlings from separator 18, in combination with the previously specified rates, maintains the middlings at a viscosity of about 1.5 centipoises and a density of about 1.14. From the flotation cell additional oil froth is obtained in amount of about 7 lbs./hr. and about 105 lbs./hr. of oil-lean middlings are withdrawn therefrom and discarded. Use of the flotation cell increases the recovery of oil from the tar sands to an overall value of about 85%.

In the foregoing specifically described embodiment of the invention the density and viscosity of the oil-rich

middlings layer remain approximately at the values stated as long as the tar sands feed composition remains as specified. Whenever the content of silt and clay in the feed changes substantially, the fresh water addition rate and the oil-rich middlings withdrawal rate are adjusted accordingly to maintain the density and/or viscosity at the desired levels as previously specified.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. Process for separating oil from bituminous sands which comprises:

(a) forming a mixture of the bituminous sands and water including that from the hereinafter specified recycle stream, the amount of water in said mixture being 0.2-3.0 lbs./lb. of the bituminous sands;

(b) passing the mixture into a separation zone;

(c) settling the mixture in the separation zone at a temperature in the range of 130-210°F. to form an upper oil froth layer, a middlings layer comprising water, clay and oil, and a sand tailings layer;

(d) separately removing the oil froth layer and the sand tailings layer;

(e) removing a stream of middlings layer from the separation zone and utilizing the same as the aforesaid recycle stream in forming said mixture in Step (a);

(f) passing a second stream of middlings layer to a scavenger zone and therein recovering an additional amount of oil froth;

(g) regulating the amount of water incorporated with said bituminous sands in Step (a) and the rate of passage of said second stream to the scavenger zone in Step (f) so as to regulate and maintain the density of said middlings layer within the range of 1.03-1.50 g./cc. and the viscosity thereof within the range of 0.5-10 centipoises; and

(h) removing from the scavenger zone and discarding from the system middlings material of depleted oil content comprising clay dispersed in water.

2. Process according to claim 1 wherein said temperature is at least 170°F., said viscosity is maintained within the range of 0.6-3.0 centipoises, and the recovery of additional oil in Step (f) is effected by air flotation.

3. In a process for separating oil from bituminous sands which process comprises forming a pulp of the bituminous sands with a minor amount of water in a pulping zone; removing pulp therefrom and mixing the same with hot water and a hereinafter specified recycle stream in a dilution zone; flushing the mixture from the dilution zone into a separation zone, settling the mixture in the separation zone at a temperature in the range of 130° to 210°F. to form an upper oil froth layer, a middlings layer comprising water, clay and oil, and a sand tailings layer; separately removing the oil froth layer and the sand tailings layer; removing a stream of middlings layer from the separation zone and passing it to the dilution zone as the aforesaid recycle stream; and passing a second stream of middlings layer to a separate recovery zone; the improvement which comprises regulating the amount of water incorporated with said bituminous sands and the rate of passage of said second stream to said separate recovery zone so as to regulate and maintain the density of said middlings layer within the range of 1.03 to 1.50 g./cc.

4. Process according to claim 3 wherein the amount of water present in the mixture formed from the pulp, hot water and recycle stream in said dilution zone is 0.2 to 3.0 lbs./lb. of said bituminous sands.

5. Process according to claim 4 wherein said density is maintained within the range of 1.10-1.20 g./cc. and said temperature is at least 170°F.

6. Process according to claim 5 wherein the pH of the middlings layer in said separation zone is maintained in the range of 7.5-9.0 by the addition of an alkali metal-containing alkaline reagent to said pulping zone.

7. Process according to claim 6 wherein said alkaline reagent is selected from the group consisting of caustic soda, sodium carbonate and sodium silicate.

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8. Process for separating oil from bituminous sands which comprises:

(a) forming a pulp of the bituminous sands with a minor amount of water in a pulping zone;

(b) removing pulp therefrom and mixing the same with hot water and the hereinafter specified recycle stream in a dilution zone;

(c) flushing the mixture from the dilution zone into a separation zone;

(d) settling the mixture in the separation zone at a temperature in the range of 130-210°F. to form an upper oil froth layer, a middlings layer comprising water, clay and oil, and a sand tailings layer;

(e) separately removing the oil froth layer and the sand tailings layer;

(f) removing a stream of middlings layer from the separation zone and passing it to the dilution zone as the aforesaid recycle stream;

(g) passing a second stream of middlings layer to a separate recovery zone and therein subjecting it to air flotation to recover an additional amount of oil;

(h) regulating the amount of water incorporated with said bituminous sands and the rate in Step (g) of passage of said second stream to said separate recovery zone so as to regulate and maintain the viscosity of said middlings layer within the range of 0.5-10 centipoises; and

(i) removing from said separate recovery zone middlings material of depleted oil content comprising clay dispersed in water.

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9. Process according to claim 8 wherein the amount of water present in the mixture in Step (b) is 0.2-3.0 lbs./lb. of said bituminous sands.

10. Process according to claim 9 wherein said viscosity is maintained within the range of 0.6-3.0 centipoises and said temperature is at least 170°F.

11. Process according to claim 10 wherein the pH of the middlings layer in said separation zone is maintained in the range of 7.5-9.0 by the addition of an alkali metal-containing alkaline reagent to said pulping zone.

12. Process according to claim 11 wherein said alkaline reagent is selected from the group consisting of caustic soda, sodium carbonate and sodium silicate.

13. Process for separating oil from bituminous sands which comprises:

(a) forming a mixture of the bituminous sands and water such that the amount of water in said mixture is 0.2-3.0 lbs./lbs. of the bituminous sands;

(b) passing the mixture into a separation zone;

(c) settling the mixture in the separation zone at a temperature in the range of 130-210°F. to form an upper oil froth layer, a middlings layer comprising water, clay and oil, and a sand tailings layer;

(d) separately removing the oil froth layer and the sand tailings layer;

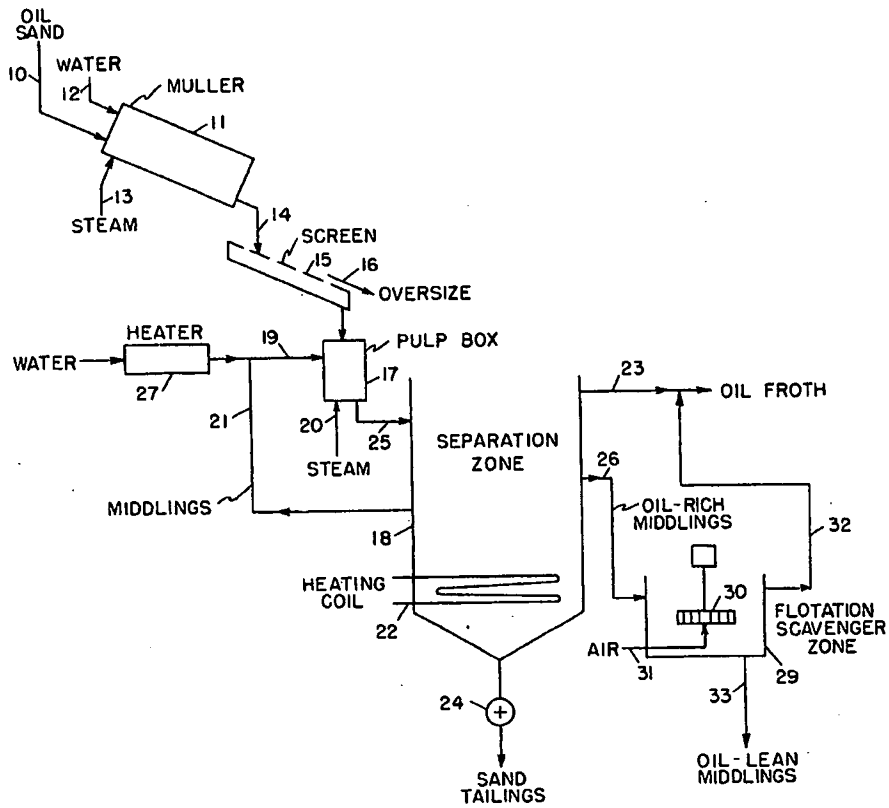
(e) passing a stream of middlings layer from the separation zone to a scavenger zone and therein recovering an additional amount of oil froth;

(f) regulating the amount of water incorporated with said bituminous sands in Step (a) and the rate of passage of said stream to the scavenger zone in Step (e) so as to regulate and maintain the density of said middlings layer within the range of 1.03-1.50 g./cc. and the viscosity thereof within the range of 0.5-10 centipoises; and

(g) removing from the scavenger zone and discarding from the system middlings material of depleted oil content comprising clay dispersed in water.

14. Process according to claim 13 wherein said temperature is at least 170°F., said viscosity is maintained within the range of 0.6-3.0 centipoises, and the recovery of additional oil in Step (e) is effected by air flotation.

15. Process according to claim 13 wherein a stream of middlings layer, other than the stream specified in Step (e), is recycled toStep (a) for use in forming said mixture of bituminous sands and water.



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